

**SOLID COMPOSITION WITH AN AQUEOUS CONTINUOUS PHASE,  
COMPRISING A HYDROPHILIC GELLING AGENT AND A PARTICULAR  
FILLER, AND USES THEREOF**

5           The present invention relates to a solid composition with an aqueous continuous phase, as well as to its use in cosmetics, especially for making up the skin and/or mucous membranes and/or keratin fibres.

10           Products which are in solid form are known in the cosmetics industry. Products of this type which may be mentioned, for example, in the make-up field include tubes or "sticks" of lipstick, of foundation or of eyeshadow; in the field of skincare or lipcare include lip repair pencils and depigmenting, make-up-removing  
15 or moisturizing tubes or "sticks"; in the field of hygiene include deodorant sticks and foam sticks or bars for shaving or washing the skin.

          In point of fact, it is particularly advantageous to have available products in stick form  
20 since such products are very practical to use, easy to transport and there is no risk of the product running.

          Moreover, make-up products are generally formulated on the basis, on the one hand, of a fatty phase for reasons of comfort and softness, and, on the  
25 other hand, of a pulverulent phase which gives the desired colour. This pulverulent phase may comprise pigments and/or fillers and/or nacres. The fatty phase

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generally comprises waxes and/or pasty compounds which give the sticks structure.

However, sticks formulated with a wax base have certain drawbacks: they have a greasy nature which  
5 users do not find appealing and they lack freshness when applied. In addition, it is difficult to introduce hydrophilic active agents therein.

It is therefore increasingly being sought to make make-up sticks comprising an aqueous phase in the  
10 highest concentration possible. However, sticks comprising a large aqueous phase are occasionally subject to problems of stability and lack of cohesion. In particular, these gels, which are made from a combination of a hydrophilic gelling agent and water,  
15 have the drawback of being fragile and of breaking easily during use.

One means for improving the gel strength is to increase the concentration of hydrophilic gelling agent, but the gels then become difficult to  
20 disintegrate, i.e. the amount of material lifted during uptake of the product is insufficient.

Moreover, aqueous sticks that do not contain a fatty phase give rise to sensations of drying out and of tautness of the skin when applied, and these  
25 sensations are considered undesirable by users.

Thus, the need remains for a solid composition which may be used by direct application to

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the skin or using a sponge, which disintegrates well, is comfortable and gives a fresh sensation when applied without an effect of tautness or drying out.

The present invention thus relates to a solid  
5 composition with an aqueous continuous phase comprising  
i) at least one hydrophilic gelling agent and ii) at  
least one pulverulent phase comprising at least one  
lamellar filler.

The compositions according to the invention  
10 are particularly comfortable: they apply easily and  
give a sensation of freshness and softness. They do not  
cause any sensation of tautness or drying out of the  
skin after application. They have excellent cosmetic  
properties.

15 The compositions of the invention have  
excellent application and disintegration qualities. In  
particular, by virtue of the combination according to  
the invention, the level of disintegration obtained,  
for an equivalent hardness, is superior to that of the  
20 known sticks. The product is easy to take up, and this  
can be done directly onto the body or with the fingers  
or a sponge, by taking a sufficient amount of product,  
and it is easy subsequently to apply to the skin  
homogeneously, without needing to be moistened  
25 beforehand. The make-up effect obtained is uniform and  
homogeneous.

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These compositions moreover have excellent cohesion. They are stable over time and heat-stable. Thus, after they have been stored for two months at ambient temperature or at 45°C, they show no phenomenon  
5 of syneresis (exudation) or of phase separation: their appearance and hardness remain unchanged.

The compositions according to the invention do not exude, even at low contents of gelling agent, and they do not require mandatory intervention of a  
10 particular preparation technique.

A subject of the present invention is also a make-up product for the skin or keratin fibres, comprising a composition as defined above.

A subject of the present invention is also a  
15 process for making up the skin and/or mucous membranes and/or keratin fibres, which consists in applying to the latter a solid composition and/or a solid make-up product as defined above.

For the purposes of the present invention,  
20 the expression "solid composition" means a composition with a hardness, defined by a maximum force before breaking, ranging from 5 to 130 grams, at ambient temperature (20-25°C), after penetration with a stainless steel spindle 2 mm in diameter into the  
25 matrix of the composition to a depth of 1 mm at a speed of 1 mm/s and removal of the said spindle from the matrix of the composition at a speed of 2 mm/s; the

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maximum force before breaking being measured using a texture analyser such as a "TAXT2" machine sold by the company Rheo.

The composition according to the invention comprises a hydrophilic gelling agent. The term "gelling agent" means a compound which, in the presence of a solvent, creates more or less strong intermacromolecular bonds thus inducing a three-dimensional network which sets the said solvent.

This hydrophilic gelling agent may be chosen from polysaccharides, protein derivatives, synthetic or semi-synthetic gels of polyester type, in particular of sulphonic polyester type, and polyacrylates or polymethacrylates, and derivatives thereof.

Among the polysaccharides which may be mentioned are:

- algal extracts such as agar-agar, carrageenans (iota, kappa or lambda carrageenan) and alginates, in particular sodium or calcium alginate;
- microorganism exudates such as xanthan gum and its derivatives, for instance the product sold under the trade name "Rheosan" by the company Rhodia Chimie, and gellan;
- fruit extracts such as pectins;
- gelling agents of animal origin, such as protein derivatives, in particular bovine or fish gelatin, and caseinates;

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- polysaccharides containing a side chain and 6 neutral sugars, as disclosed in document FR-A-2 759 377;
- and mixtures thereof.

The hydrophilic gelling agent is preferably  
5 chosen from polysaccharides, and even more preferably the hydrophilic gelling agent is gellan.

As products that are particularly suitable for the invention, mention may be made of the gellan gum sold under the trade name "Kelcogel F" by the  
10 company Nutrasweet-Kelco or the iota carrageenan sold under the trade names "Seaspen PF 357" or "Viscarin SD 389" by the company FMC.

The hydrophilic gelling agent is present in the composition according to the invention at a  
15 concentration ranging up to 20% by weight and preferably from 0.2% to 10% by weight relative to the total weight of the composition. This concentration makes it possible to obtain a hardness and consistency that are suitable for ideal disintegration.

20 The composition according to the invention also comprises a pulverulent phase which may comprise at least one lamellar filler.

The lamellar filler used according to the invention may be in the form of particles with a mean  
25 size of greater than or equal to 5 microns, preferably ranging from 10 microns to 300 microns and in particular ranging from 10  $\mu\text{m}$  to 40  $\mu\text{m}$ .

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The lamellar filler maybe of mineral origin and may be chosen, for example, from phyllosilicates.

Lamellar fillers which may be used in particular are:

- 5 - talc, which is a hydrated magnesium silicate, and in particular those sold under the names "Talc Luzenac 00" by the company Luzenac, "Talc P3" by the company Nippon Talc;
- kaolin, which is a hydrated aluminium silicate in the form of particles of anisotropic form which are generally less than 30  $\mu\text{m}$  in size; kaolins which may be used include the product sold under the name "Kaolin Supreme 1" from English China Clays,
- boron nitride, and in particular the products sold under the names "Ceram Blanche 1" and "Ceram Blanche" by the company SPCI;
- mica, or aluminosilicate, which may be chosen from muscovite, phlogopite, tiotite, sericite, lepidolite, paragonite, margarite, roscoelite, artificial or synthetic mica with a fluorine atom replacing the hydroxyl group of natural mica, as well as fired or calcined products of these micas. The micas are generally in the form of flakes from 2 to 200  $\mu\text{m}$  and preferably 5-70  $\mu\text{m}$  in size and from 0.1 to 5  $\mu\text{m}$  and preferably 0.2-3  $\mu\text{m}$  in thickness. Micas which may be used, for example, are those sold under the names "Mica SFG70" by the company Aspanger and "Mica Concord 1000"

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- by the company Sciama; the product sold under the trade name "Cashmir K 2" by Catalysis and Chemicals, which is a mica with a mean particle size of greater than 15 microns coated with silica beads with a mean
- 5 particle size of 0.3 microns, may also be used;
- natural mother-of-pearl, mica coated with titanium oxide, with iron oxide, with natural pigment or with bismuth oxychloride, as well as coloured titanium mica.
- Mention may be made in particular of titanium micas
- 10 such as the nacres "Timica Golden Bronze 240/A" from Engelhard or "Colorona Red Gold" from Merck,
- lamellar silica such as, in particular, the products sold under the names "SG Flake 3 M" by the company Maprecos or "Chemicelen" by the company Sumitomo;
- 15 - lamellar titanium oxide, iron oxide or zinc oxide;
- bismuth oxychloride;
  - lauroyllysine;
  - molybdenum sulphide;
  - and mixtures thereof.

20 Preferably, fillers which have a satin or gloss glint are chosen, which makes it possible to avoid the dull and rather matt appearance which develops on drying.

Preferably, the lamellar filler is chosen

25 from boron nitride, mica, mica coated with silica beads, natural mother-of-pearl, mica coated with titanium oxide, with iron oxide, with natural pigment

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or with bismuth oxychloride, and coloured titanium mica.

The lamellar filler may be present in the composition according to the invention in a content  
5 ranging from 0.1% to 50% by weight and more preferably from 0.5% to 20% by weight relative to the total weight of the composition.

The pulverulent phase may comprise, besides the lamellar fillers mentioned above, other fillers,  
10 which may be mineral or synthetic, and also pigments.

Other fillers which may be mentioned include silica, Nylon powder, polyethylene powder, Teflon, starch, tetrafluoroethylene polymer powders, polymethyl methacrylate powders, polyurethane powders, polystyrene  
15 powders, polyester powders, synthetic hollow microspheres, undeformable silicone resin microbeads, zinc oxide, titanium oxide, zirconium oxide, cerium oxide, precipitated calcium carbonate, magnesium carbonate, magnesium hydrocarbonate, hydroxyapatite,  
20 hollow silica microspheres, glass or ceramic microcapsules, metal soaps derived from carboxylic organic acids containing from 8 to 22 carbon atoms and preferably from 12 to 18 carbon atoms, for instance zinc, magnesium or lithium stearate, zinc laurate or  
25 magnesium myristate,  $\text{SiO}_2/\text{TiO}_2/\text{SiO}_2$ ,  $\text{TiO}_2/\text{CeO}_2/\text{SiO}_2$  or  $\text{TiO}_2/\text{ZnO}/\text{talc}$  compounds, and polyethylene

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terephthalate/polymethacrylate polymers in the form of flakes.

The fillers other than the lamellar fillers may be present in the composition in a proportion of 0.1-60% by weight, relative to the total weight of the composition, preferably in a proportion of from 0.1 to 40% and more preferably 1-20%.

The term "pigment" should be understood as meaning white or coloured, mineral or organic particles that are insoluble in the medium, and that are intended to colour and/or opacify the composition.

The pigments may be present in a proportion of 0-40% by weight, relative to the total weight of the composition, preferably in a proportion of from 0.1% to 30% and more preferably in a proportion of 1-20%. They may be white or coloured, mineral and/or organic, and of typical or nanometric size. The term "nanometric size" means pigments whose mean particle size ranges from 5 nm to 100 nm.

Among the mineral pigments and nanopigments which may be mentioned are titanium dioxide, zirconium dioxide or cerium dioxide, as well as zinc oxide, iron oxide or chromium oxide, nanotitanias and ferric blue. Among the organic pigments which may be mentioned are carbon black and the lakes commonly used to give the lips and the skin a make-up effect, which are calcium,

barium, aluminium or zirconium salts, and acidic dyes such as halo-acid dyes, azo dyes or anthraquinone dyes.

The pigments may be coated in particular with silicone compounds such as PDMSs and/or with polymers, 5 in particular polyethylenes, or alternatively with fluoro compounds. Mention may thus be made of the SA pigments from Maprecos or the PI pigments from Myoshi.

The compositions according to the invention may also comprise a floral water such as cornflower 10 water and/or a mineral water such as eau de Vittel, eau de Lucas or eau de La Roche Posay and/or a thermal spring water.

The compositions according to the invention may also comprise water-soluble dyes chosen from the 15 dyes commonly used in the field under consideration, such as the disodium salt of ponceau, the disodium salt of alizarin green, quinoline yellow, the trisodium salt of amaranth, the disodium salt of tartrazine, the monosodium salt of rhodamine, the disodium salt of 20 fuchsin and xanthophyll.

Preferably, the compositions according to the invention comprise up to 99.95% by weight and preferably from 30% to 99.5% by weight of water, relative to the total weight of the composition.

25 The compositions according to the invention may also comprise solvents other than water, such as, for example, primary alcohols such as ethanol and

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isopropanol, glycols such as propylene glycol, butylene glycol, dipropylene glycol and diethylene glycol, and glycol ethers such as the mono-, di- or tripropylene glycol C<sub>1</sub>-C<sub>4</sub> alkyl ether and mono-, di- or triethylene glycol, and mixtures thereof.

The compositions according to the invention may also comprise a fatty phase which may comprise at least one oil. For the purposes of the present invention, the term "oil" means a fatty substance which is liquid at ambient temperature (25°C).

Among the oils which may be used, mention may be made of oils of animal, plant or mineral origin, such as liquid paraffin, liquid petroleum jelly, perhydrosqualene, apricot oil, wheatgerm oil, sweet almond oil, beauty-leaf oil, sesame oil, macadamia oil, grape pip oil, rapeseed oil, coconut oil, groundnut oil, palm oil, castor oil, avocado oil, jojoba oil, olive oil or cereal germ oil; fatty acid esters of polyols, in particular liquid triglycerides; alcohols; acetylglycerides; alkyl or polyalkyl octanoates, decanoates or ricinoleates; fatty acid triglycerides; glycerides, fluoro oils and perfluoro oils; synthetic oils such as fatty esters; silicone oils such as volatile silicone oils, polymethylsiloxanes, polymethylphenylsiloxanes, polysiloxanes modified with fatty acids, with fatty alcohols or with

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polyoxyalkylenes, fluorosilicones and perfluoro oils, and mixtures thereof.

The fatty phase may be present in proportions ranging, for example, up to 70% and preferably from 5% to 50% by weight relative to the total weight of the composition.

The compositions according to the invention may also be in the form of oil-in-water (O/W) emulsions. In this case, they may comprise an O/W surfactant system with an HLB (hydrophilic/lipophilic balance) of greater than or equal to 7, which are usually used in cosmetics. O/W surfactant systems which may be mentioned in particular (CTFA) are:

cetearylglucoside, sucrose stearate, PEG-40 stearate, sorbitan tristearate, sorbitan stearate, polysorbate 60, sorbitan stearate/sucrose cocoate mixture, glyceryl stearate/PEG-100 stearate mixture, PEG-400, glyceryl stearate, and PEG-6/PEG-32/glycol stearate mixture, and mixtures thereof.

Mention may be made in particular of the mixture of glyceryl stearate/PEG-100 stearate sold under the trade name "Arlacel 165 FL" by the company Unichema, the sorbitan monostearate oxyethylenated with 20 EO (ethylene oxide) sold under the trade name "Polysorbate 60" by the company Unichema, the polyethylene glycol monostearate 8 EO sold under the trade name "Estol 3646" by the company Unichema, and

the sucrose mono-dipalmitostearate sold under the trade name "Tegosoft PSE 141 G" by the company Goldschmidt.

The surfactant system is preferably present in the compositions according to the invention in a content ranging from 0.1% to 15% and preferably from 0.5% to 7% by weight relative to the total weight of the composition.

It is possible to modify the rigidity of the compositions according to the invention by adding thereto one or more salts which will increase this rigidity. These salts may be chosen from mono-, di- and trivalent metal salts, and more particularly alkali metal and alkaline-earth metal salts, and in particular sodium, calcium or magnesium salts. The ions constituting these salts may be chosen, for example, from carbonates, bicarbonates, sulphates, glycerophosphates, borates, chlorides, nitrates, acetates, hydroxides and persulphates, and also the salts of  $\alpha$ -hydroxy acids (citrates, tartrates, lactates or malates) or of fruit acids, or alternatively amino acid salts (aspartate, arginate, glycocholate or fumarate). The amount of salt may range from 0.01% to 2% and preferably from 0.1% to 1% relative to the total weight of the emulsion.

The salt is preferably chosen from calcium, magnesium or strontium nitrate, calcium or magnesium borate, calcium, sodium, magnesium, strontium,

neodymium or manganese chloride, magnesium or calcium sulphate and calcium or magnesium acetate, and mixtures thereof. More preferably, the salt is chosen from magnesium chloride and sodium chloride.

5           The compositions of the invention also contain a cosmetically or physiologically acceptable medium, i.e. a medium which is compatible with all keratin materials such as the skin, the nails, the hair, the eyelashes, the eyebrows, the mucous membranes  
10 and the semi-mucous membranes, and any other area of body or facial skin.

          The composition may also comprise any additional compound usually used in cosmetics. These additional compounds may be chosen from antioxidants,  
15 fragrances, essential oils, preserving agents, lipophilic or hydrophilic cosmetic or pharmaceutical active agents, moisturizers, vitamins, essential fatty acids, sphingolipids, self-tanning compounds such as DHA, and sunscreens, and mixtures thereof.

20           Needless to say, a person skilled in the art will take care to select this or these optional additional compound(s), and/or the amount thereof, such that the advantageous properties of the composition according to the invention are not, or not  
25 substantially, adversely affected by the addition envisaged.

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The compositions according to the invention may be prepared according to the conventional methods for preparing cosmetic compositions, in particular O/W gels and emulsions, these methods being well known to those skilled in the art.

The compositions according to the invention may constitute make-up products or care products for the skin, in particular body or facial skin and/or the scalp, or for keratin fibres, in particular the hair, the nails, the eyelashes and/or the eyebrows, or alternatively for the mucous membranes, in particular the lips. They may thus constitute make-up products for the body, foundations, eyeshadows, face powders, concealers, lipsticks, lip contour pencils, mascaras, eye contour pencils and sticks for dyeing or making up locks of hair.

The invention is illustrated in greater detail in the examples which follow.

In the examples which follow, the amounts are given as percentages by weight relative to the total weight of the composition.

**EXAMPLE 1:**

The Applicant prepared the following body tattoo stick:

- Gellan gum sold under the trade name "Kelcogel F" from Nutrasweet-Kelco 0.5%
- Starch: amylopectin/amylose crosslinked with



epichlorohydrin, sold under the trade name "non-mucilaginous insoluble rice starch" by Rémy 4%

- Bronze-coloured nacres:

"Timica Golden Bronze 240/A" from Engelhard 3%

5 "Colorona Red Gold" from Merck 2%

-  $\text{MgCl}_2$  0.1%

- Preserving agents 0.75%

- Water qs 100%

Preparation: a gel of gellan in water is formed by

10 heating at 80°C for 15 min. Next, the starch is incorporated at 70°C for 20 min, followed by the salt. After waiting for 10 min, the nacres are then added. After 5 min, the mixture is cast while hot.

This stick has a hardness, measured as  
15 described above, of 24 g  $\pm$  2.5 g.

It disintegrates very easily and makes it possible to produce precise "tags" on the body.

#### **EXAMPLE 2:**

The Applicant prepared the following  
20 foundation:

- Gellan gum sold under the trade name

"Kelcogel F" from Nutrasweet-Kelco 0.5%

- Water qs 100%

- Preserving agent qs

25 - NaCl 0.1%

- Mica (15 microns) coated with silica beads  
(0.3 microns) (97/3) sold under the trade name

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- "Cashmir K II" from Catalysis and Chemicals 3%
- Starch: amylopectin/amylose crosslinked with  
epichlorohydrin, sold under the trade name "non-  
mucilaginous insoluble rice starch" by Rémy 4%
- 5 - Iron oxide pigments 7%
- Glycerol 7%

This stick was prepared in the same manner as  
in Example 1.

It has a hardness, measured as described  
10 above, of  $28.5 \pm 2.5$  g.

This stick disintegrates well and has a  
luminous effect.

**EXAMPLE 3:**

The Applicant prepared the same stick as in  
15 Example 2, but replacing the "Cashmir K 2" with 4%  
boron nitride: the stick obtained has a soft and silky  
effect and disintegrates well.

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